

Reference Data for the Thermal Conductivity of Saturated Liquid Toluene Over a Wide Range of Temperatures

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Efficient design of industrial processes and equipment requires accurate thermal conductivity data for a variety of fluids, such as alternative refrigerants, fuels, petrochemicals, aqueous systems, molten salts, and molten metals. The accuracy of experimental thermal conductivity data is a function of the operating conditions of the instrument. Reference data are required over a wide range of conditions to verify the claimed uncertainties of absolute instruments and to calibrate relative instruments, since either type may be used to measure the thermal conductivity of fluids. Recently, accurate experimental data for the thermal conductivity of liquid toluene near the saturation line have been obtained, which allow the upper temperature limit of the previous reference-data correlation to be extended from 360 to 553 K. The thermal conductivity was measured using two transient hot-wire instruments from 300 to 550 K, the first with a bare 12.7 μm platinum wire and the second using an anodized 25 μm tantalum wire. Uncertainties due to the contribution of thermal radiation and the purity of the samples are discussed. The proposed value of the thermal conductivity of liquid toluene at 298.15 K and 0.1 MPa is $0.13\,088 \pm 0.000\,85$. The quality of the data is such that new improved recommendations and recommended values can be proposed with uncertainties at 95% confidence of 1% for $189 < T < 440$ K, 1.5% for $440 < T < 480$ K, and 2% for $480 < T < 553$ K, near the saturation line. © 2000 American Institute of Physics. [S0047-2689(00)00102-1]

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1. Introduction

In recent papers,¹⁻³ reference data and correlations have been established for the thermal conductivities of toluene, water, benzene, and *n*-heptane over most of their liquid range at saturation. To establish these reference data and correlations, the data in the literature were assessed by a careful analysis of the experimental methods, sample purity, estimated uncertainty, and equipment used, then designated as primary or secondary data. The primary data were used to develop correlations for the thermal conductivities of these saturated liquids as functions of temperature. This effort¹⁻³ was carried out under the auspices of the Subcommittee on Transport Properties of the Commission on Thermodynamics of the International Union of Pure and Applied Chemistry; subsequently these correlations, for toluene, water, and *n*-heptane, were included as a part of a complete set of recommendations on the physical-chemical properties of fluids by IUPAC.⁴

Toluene can be obtained with very high purity and is a liquid over a very wide range of temperature. Thus, toluene is a good reference material for many thermophysical properties. Its thermal conductivity along the saturation line varies by a factor of 2 from 180 to 553 K. Since our previous publications,^{1,4} new absolute data have been published for toluene⁵⁻¹³ which have reduced the uncertainty and extended the temperature range of the available data, so it is now possible to improve the correlations proposed earlier. Extensive studies of the influence of radiative heat transport in transient hot-wire instruments have also been reported.^{14,15} These studies have shown that the transient hot-wire method is much less affected by thermal radiation than steady-state

techniques are. The more recent analysis demonstrates that the results previously reported up to 370 K^{1,2} were not affected by thermal radiation, at least within their estimated uncertainties. Furthermore, this study indicates that even at high temperatures, where the energy transport due to infrared radiation becomes comparable to, or exceeds, the uncertainty of the data, it can be accounted for by using a correction developed by Menashe and Wakeham¹⁵ and Nieto de Castro *et al.*¹⁶

This paper presents improved correlations based on revised reference data for the thermal conductivity of toluene between 189 and 553 K. The earlier work relied only on data obtained with bare transient hot-wire instruments. It is preferable that reference data be obtained with multiple experimental techniques. In the present work, primary data are available from transient hot-wire measurements with bare and coated wires. Light-scattering measurements of thermal diffusivity are also available, which allow the absence of thermal-radiation errors to be verified at the highest temperatures. Data with both bare and coated wires are available from 250 to 553 K. Only the region from 178.15 K (triple point) to 250 K is based solely on bare transient hot-wire measurements, but here data are available from two laboratories. The uncertainty at the lowest temperatures is significantly reduced due to the new data of Yamada *et al.*⁹ Extensive measurements of the thermal conductivity of saturated liquid toluene were made at temperatures from 300 to 553 K using two transient hot-wire instruments at NIST, one with a bare platinum wire⁷ and the second using an anodized tantalum wire.¹⁰ The uncertainties of the present correlations is smaller than those of the earlier recommendations^{1,4} in the overlapping temperature range, and the upper temperature limit of the correlation has been extended from 370 to 553 K, nearly to the critical temperature (593.95 K).

2. Experimental Techniques

The experimental methods used for the measurement of thermal conductivity can be divided into two groups: steady state and transient. The thermal conductivity can also be obtained indirectly from light-scattering measurements of the thermal diffusivity, a technique which is particularly valuable near the critical region and at high temperatures. For an overall discussion of the experimental techniques for the measurement of the thermal conductivity of liquids the reader is referred to recent monographs on the subject.^{17,18}

The transient hot-wire technique is accepted as the most accurate method for the measurement of the thermal conductivity of electrically nonconducting fluids, with the exceptions of regions of the phase diagram in the vicinity of the critical point and at very low densities in the gaseous phase.¹⁸ However, a large number of fluids are electrically conducting, including polar liquids such as water, refrigerants, inorganic melts, electrolyte solutions, molten metals, and molten semiconductors. Instruments using the transient hot-wire technique must be modified to study these types of fluids. One popular alternative is to electrically insulate tantalum hot wires from the fluid with a thin coating of Ta₂O₅

TABLE 1. Primary sources of experimental data for thermal conductivity of liquid toluene

Literature source	Technique ^{a,b}	Temperature range (K)	No. of data points	Assigned uncertainty (\pm %)	Purity of samples used ^c
Pitmann 1968 ²⁷	THW	181–326	50	1.5	MPNP
Nieto de Castro <i>et al.</i> 1977 ²⁴	THW	291–323	12	1.0	MPFP,D
Nagasaka and Nagashima 1981 ^{25,26}	THW	274–355	5	1.0	MPNP
Nieto de Castro <i>et al.</i> 1983, ¹⁶ 1985 ²⁸	THW	308–360	5	1.0	DGSA
Charitidou <i>et al.</i> 1987 ¹¹	THW	307–346	12	0.5	MPFD
Taxis <i>et al.</i> 1988 ¹²	THW	308–347	6	0.5	PDSA
Ramires <i>et al.</i> 1989 ⁵	THW	300–367	11	0.5	PDSA
Richard and Shankland 1989 ¹³	THW	311–343	4	0.9	MPNP
Perkins <i>et al.</i> 1991 ⁷	THW	300–480	8	0.5	PDSA
Perkins <i>et al.</i> 1991 ⁷	THW	480–553	3	1.5	PDSA
Assael <i>et al.</i> 1992 ⁸	TCHW	250–335	12	0.5	MPNP
Ramires <i>et al.</i> 1993 ⁶	TCHW	299–368	37	0.5	PDSA
Yamada <i>et al.</i> 1993 ⁹	THW	189–393	11	0.5	MPNP
Perkins <i>et al.</i> 1999 ¹⁰	TCHW	369–480	5	0.5	PDSA
Perkins <i>et al.</i> 1999 ¹⁰	TCHW	480–550	2	1.5	PDSA

^aTHW—transient hot wire technique.^bTCHW—transient coated hot wire technique.^cAcronyms defined in Sec. 3.2.

produced by anodization. A comprehensive discussion of this problem has been presented¹⁹ and recently described in a reference-data publication on water.³ The thermal conductivity data considered in the present work were obtained with transient hot-wire instruments using hot wires of both bare platinum and anodized tantalum.

3. Experimental Data

In this work we use the recommendations previously followed¹ for the selection of primary and secondary data. The primary data were identified using several criteria as explained in Assael and co-workers.² Due to the quality of the new experimental data, the upper limit that was established for the uncertainty of the primary data was 1%, except in the lower and upper temperature extremes, where some points with 1.5% estimated uncertainty were used. All uncertainties are expressed with a confidence interval of 95% (2σ).

3.1. Purity of Samples Used

Toluene is known to be a very good solvent for nonpolar organic materials and is also a hygroscopic material. It is therefore important to consider the purities of the samples used by the different authors, especially those that contribute to the primary data sets. The first consistent analysis of the effect of impurities on the thermal conductivity of organic liquids was made by Poltz and co-workers.^{20–23} These authors devised purification procedures for toluene, carbon tetrachloride, alcohols, and dialkylphthalates based on their assessment of the contribution of radiative heat transfer on the measured thermal conductivities in steady-state parallel-plate instruments. The quantity of water was their major concern, because of its high value of thermal conductivity and its strong absorption for infrared radiation. Poltz and

co-workers^{20–23} recommended reference materials such as toluene and dimethylphthalate because of their absorption in the infrared region, and because they could be purified and dried using molecular sieves to remove water to a level of micrograms per gram.

3.2. Primary Data

A summary of primary data for toluene, together with their estimated uncertainty and purity classification, is given in Table 1. The purification of the samples used by the different authors for the data selected as primary was classified as:

MPNP—manufacturers stated purity, no further purification;

MPFP—manufacturers stated purity, further purification; D—dried with molecular sieves, sodium metal, or calcium hydride;

DGSA—degassed sample, GC analysis;

PDSA—purified and dried samples with GC or GC-MS analysis.

For toluene, the purities were in excess of 99.7% for nonpurified samples and better than 99.95% for purified and dried samples.

The statistical treatment of data is the same as that which was adopted in previous work.^{1–3} In Table 1 we have maintained the uncertainties which were previously assigned to the older data sets.^{16,24–28} For the new data sets,^{5–13} the assigned uncertainty is that claimed by each author. Since a temperature rise is required for thermal conductivity determinations, it is not possible to measure the thermal conductivity precisely at saturation. Thermal conductivity measurements must be made at pressures which are slightly higher than the saturation pressure. The measurements used in the present work are within 0.5 MPa of the saturation pressure.

As shown before,¹⁶ a change in pressure of 1 MPa does not change the thermal conductivity of liquid toluene by more than 0.3%. In practice, for the data considered here there is no distinction between the values of the thermal conductivity at the measured pressure relative to saturation values, since the best experimental uncertainties are $\pm 0.5\%$. Many of the low-temperature ($T < 320$ K) data sets were measured at 0.1 MPa, which exceeds the saturation pressure of toluene. The difference between the thermal conductivity at the saturation line and at 0.1 MPa is less than 0.05% and insignificant for such measurements.

Some comments must be made about the data sets used. The data of Yamada *et al.*⁹ were used only up to 392 K, since the authors did not make any correction for the radiative heat transfer contribution and there is no possibility of introducing such a correction. These data, and the data presented in Perkins *et al.*^{7,10} were averaged for reference isotherms before being used in the data fit. The data of Pitmann²⁷ were used only up to 326 K, since above this temperature increasing deviations were observed relative to the other available data. The data of Perkins *et al.* with bare platinum wires⁷ and with anodized tantalum hot wires¹⁰ are assigned uncertainties of 0.5% at temperatures from 300 to 480 K, and of 1.5% at temperatures from 480 to 553 K, based on estimated uncertainties, consistency of measurements at different applied power levels, and reliability of independently measured thermal-diffusivity values after correction for effects of infrared radiation.

All the experimental data sets prior to 1990, and several subsequent data sets, made use of temperatures according to the IPTS 68 scale. The data of Ramires *et al.*⁶ and Perkins *et al.*^{7,10} were reported in terms of the ITS 90 scale. The differences in temperature scales over the temperature range of this work are never larger than 40 mK. The change in the reported thermal conductivities is never larger than $0.000\,02\text{ W m}^{-1}\text{ K}^{-1}$, which is negligible relative to the uncertainties of the reported data. The correlations presented in Sec. 4 are based on the ITS 90 temperature scale.

3.3. Secondary Data

As shown in the previous publication,¹ the deviations of 21 secondary data sets from the proposed correlation were as large as 5%. Since that report, other data sets have been reported for the thermal conductivity of liquid toluene. Shulga *et al.*²⁹ used an ac hot-wire method over the temperature region from 255 to 400 K, at pressures to 1000 MPa, with an estimated uncertainty of $\pm 1\%$. Knibbe³⁰ used a hot-single-wire instrument with very short time runs over the temperature region from 273 to 473 K, at pressures from 0 to 16 MPa, with an estimated uncertainty of 0.4%, however, there appears to be much more scatter (3%) in these data, so it was designated as secondary. Kraft *et al.*³¹ used photon correlation spectroscopy to measure the thermal diffusivity of toluene over the temperature region from 293 to 523 K with an estimated uncertainty of 2.5%. Values of the thermal conductivity were calculated from the measured thermal dif-

fusivity using calculated values of the density and isobaric heat capacity from the equation of state of Goodwin³² for toluene. More recently, Fröba *et al.*,³³ using also photon-correlation spectroscopy, measured the thermal diffusivity of toluene in the temperature range 298.15–591.15 K, with an estimated uncertainty of 2.5%. As in the case of Kraft *et al.*,³¹ the authors used the values of the density and heat capacity at constant pressure obtained from the equation of state for toluene by Goodwin³² to derive values for thermal conductivity. These derived thermal conductivity values have an uncertainty which is no better than the combined uncertainties in the measured thermal diffusivity and the calculated density and heat capacity. Thus, the light-scattering results must be designated as secondary thermal-conductivity data. Pitmann's²⁷ results above 326 K were designated as secondary in the present evaluation.

4. Correlation Procedures and Results

4.1. Equation Form

Using the convention of the International Association for the Properties of Water and Steam (IAPWS) formulation for the transport properties of the water substance developed by Sengers *et al.*³⁴ and Kestin *et al.*,³⁵ we have expressed the correlation in terms of dimensionless variables λ^* and T^* defined as

$$T^* = T / 298.15\text{ K}, \quad (1)$$

$$\lambda^* = \lambda(T) / \lambda(298.15\text{ K}, 0.1\text{ MPa}), \quad (2)$$

where $\lambda(298.15\text{ K})$ is the adopted reference value for the thermal conductivity of saturated liquid toluene at 298.15 K and 0.1 MPa, given below. The primary experimental thermal conductivity data are correlated with a simple polynomial in terms of these dimensionless variables

$$\lambda^* = \sum_i b_i T^{*i}. \quad (3)$$

4.2. Correlation

The recommended thermal conductivity of toluene at 298.15 K and 0.1 MPa is established using the primary data over the temperature region from 230 to 370 K. This reference thermal conductivity is obtained by least-squares analysis with weights reflecting the uncertainties of the data shown in Table 1. The method adopted previously to assign these weights to the different data sets was also used here.¹ The reference thermal conductivity for toluene is $\lambda(298.15\text{ K}, 0.1\text{ MPa}) = 0.130\,88 \pm 0.000\,85\text{ W m}^{-1}\text{ K}^{-1}$. It is worth noting that the reference data in this region do not depart from the previous recommended values¹ by more than 0.17%, but have a smaller uncertainty (0.6% compared with 1%).

In the previous correlation,¹ the quadratic form of Eq. (3) was required to represent the thermal conductivity of saturated liquid toluene from its normal freezing point to 360 K. The data now extend to 553 K, and Perkins *et al.*⁷ have dem-

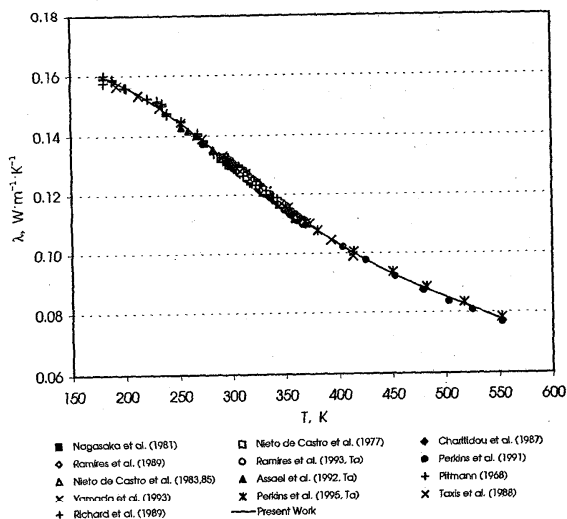


Fig. 1. The primary data selected for the thermal conductivity of toluene along the saturation line, for $189 < T < 553$ K. The line represents the correlation given by Eq. (4).

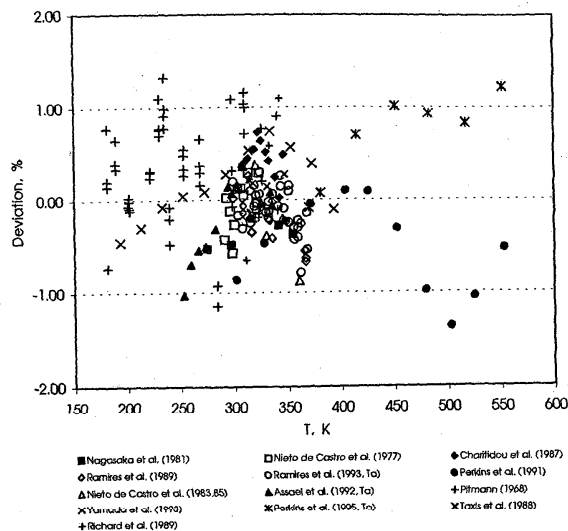


Fig. 2. Deviations of the primary data for toluene from Eq. (4).

onstrated that a similar equation is necessary for $T > 300$ K. By including the data of Yamada *et al.*,⁹ which have a smaller uncertainty than the previous data of Pitmann,²⁷ it is possible to extend the temperature range down to the normal freezing point with a single correlation equation. A fourth-order polynomial is required to represent the data over the entire temperature range from 189 to 553 K. The reduced thermal conductivity is given by

$$\lambda^* = 0.420\,919 + 3.629\,457\,T^* - 5.348\,298\,T^{*2} + 2.818\,948\,2\,T^{*3} - 0.519\,700\,T^{*4}, \quad (4)$$

for $189\text{ K} \leq T \leq 553\text{ K}$. The primary data are plotted along with this correlation in Fig. 1. The maximum deviation of the primary experimental data from Eq. (4) is 1.3%, and the standard deviation is $0.000\,62\text{ W m}^{-1}\text{ K}^{-1}$. Deviations of the primary data from the correlation represented by Eq. (4) are shown in Fig. 2.

Figure 3 shows the deviations of the more recent secondary data from the correlation of Eq. (4). The data of Pitmann²⁷ are systematically higher than Eq. (4) by about 2% near 350 K, increasing to 4.1% near 400 K. The data of Shulga *et al.*²⁹ are systematically lower by 1.5%–4%. The data of Knibbe³⁰ are systematically higher than the proposed correlation, with the deviation increasing to +4% near 400 K. The scatter of the Knibbe³⁰ data are also on the order of 3%, a value much larger than the claimed uncertainty. This large scatter may be due to inadequate correction for the large effects of the finite physical properties of the wire at the extremely short times ($t < 80$ ms) of his measurements. Finally, measurements of the thermal diffusivity (α) by Kraft *et al.*³¹ and Fröba *et al.*³³ were used to obtain thermal conductivity (λ) values using the expression

$$\lambda = \alpha \rho C_p. \quad (5)$$

The density (ρ) and heat capacity (C_p) data which are required by Eq. (5) were calculated from the Goodwin equation of state.³² These data exhibit a systematic negative deviation of -2% at the lower temperatures. However, while the data of Kraft *et al.*³¹ present a decrease in the deviation downward to -4% at 553 K, the data of Fröba *et al.*³³ exhibit a more pronounced downward trend of up to -15% for temperatures greater than 450 K. It is unlikely that these deviations are due to residual thermal-radiation effects in the transient hot-wire data, since the contribution of thermal radiation is proportional to the temperature cubed. These results seem to indicate that the heat capacities predicted from the equation of state (EOS) might be overestimated, artificially increasing the deviation of the thermal conductivity calculated from the present correlation. Furthermore, Fröba *et al.*³³ believe that the different trends in the two sets of

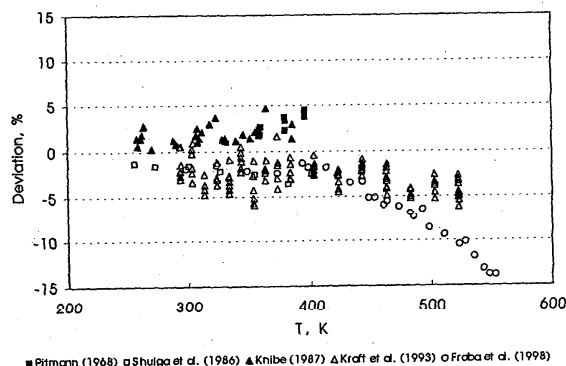


Fig. 3. Deviations of the secondary data published after 1986 for toluene from Eq. (4).

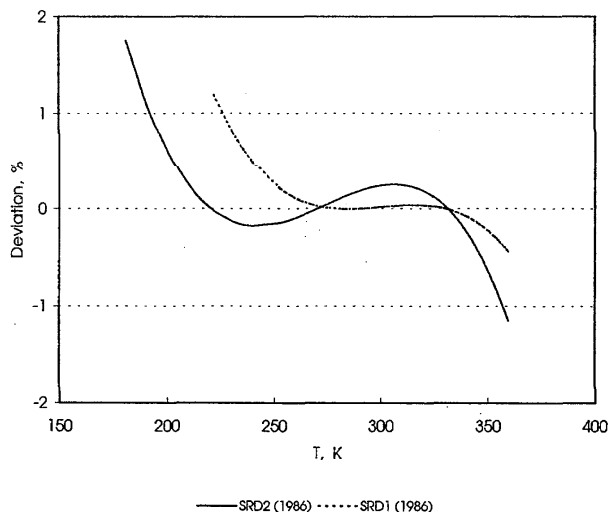


FIG. 4. Deviations of the previous reference data proposals of 1986. SRD1 is the linear correlation valid between 230 and 360 K, and SRD2 is the quadratic correlation valid between 189 and 360 K.

data, obtained with the same instrument, are due to unmeasured pressurization of the sample that occurred during the earlier measurements of Kraft *et al.*³¹ Further investigation of the heat capacity of toluene at high temperatures is necessary to resolve these differences.

4.3. Comparison with Previous Reference Correlations

Figure 4 compares the present correlation with the reference correlations of Nieto de Castro *et al.*¹ developed for 230 K $< T < 360$ K—SRD1 (1986) and 189 K $< T < 360$ K—SRD2 (1986). The maximum absolute deviations are 1.7% at the extremes of temperature, being less than 1% over most of the temperature range of the comparison.

4.4. Tabulations

Table 2 provides the recommended values for the thermal conductivity of toluene along the saturation line. The recommended values are listed to four significant digits, exceeding the uncertainty of the values. The uncertainties in the tabulated data are based on the analysis presented in Sec. 4.2. The tabulated reference data for toluene are estimated to have uncertainties of 1% for 189 K $< T < 440$ K, 1.5% for 440 K $< T < 480$ K, and 2% for 480 K $< T < 553$ K.

4.5. Cautions for the Use of the Recommended Thermal Conductivities

As emphasized in Nieto de Castro *et al.*,¹ Assael *et al.*,² and Ramires *et al.*,³ care must be taken by any user of the recommended values for the thermal conductivity. These correlations and tabulated values serve two main purposes: (i) they can be used to test the accuracy of absolute thermal-

TABLE 2. Recommended thermal conductivities for toluene

T (K)	λ (W m ⁻¹ K ⁻¹)
1%	
180	0.1588
190	0.1577
200	0.1563
210	0.1545
220	0.1525
230	0.1502
240	0.1477
250	0.1450
260	0.1423
270	0.1394
280	0.1364
290	0.1334
300	0.1304
310	0.1274
320	0.1244
330	0.1214
340	0.1185
350	0.1157
360	0.1130
370	0.1103
380	0.1078
390	0.1053
400	0.1030
410	0.1008
420	0.098 64
430	0.096 64
440	0.094 75
1.5%	
450	0.092 96
460	0.091 26
470	0.089 64
480	0.088 09
2%	
490	0.086 59
500	0.085 13
510	0.083 69
520	0.082 25
530	0.080 79
540	0.079 27
550	0.077 68

conductivity instruments, and (ii) they provide a means of calibrating relative thermal conductivity instruments.

Relative instruments should be calibrated with two reference materials over the entire temperature region of interest. Each reference fluid should preferably, have thermal conductivities and thermal radiative properties that bracket the properties of the fluid of interest. No extrapolation is required of the instrument calibration in this case. The effect of radiative heat transfer on thermal conductivity measurements is a function of the technique and the geometry of the measurement cell. The first-order heat flux due to thermal radiation is proportional to the absolute temperature cubed times the temperature gradient. Since thermal conductivity is also proportional to the temperature gradient, it is difficult to sepa-

rate thermal radiation from thermal conductivity without changes in the measurement geometry (i.e., optical path length). When the effect of thermal radiation is not correctly accounted for, it becomes a part of the apparent thermal conductivity which is measured. It has been shown that the transient hot-wire technique, if corrected properly, yields radiation-free values. Even at 553 K, the contribution of radiation for the measurements in toluene was not greater than 3.1%, and was fully accounted for by an analytical model of Nieto de Castro *et al.*¹⁶ However, this may not be the case in other instruments, especially those operating in the steady-state mode.

Toluene and water³ are recommended reference materials for instruments that can measure polar, electrically conducting liquids. Toluene and benzene² are recommended reference materials for instruments that can measure only electrically insulating liquids. The samples of the reference materials should be purified and nonaqueous materials should be dried, so that the sample purity is greater than 99.95%. It must also be verified, through the application of several different temperature gradients, that there is no fluid convection occurring during measurements of thermal conductivity.

5. Conclusions

Improved reference data are proposed for the thermal conductivity of toluene, which cover the saturated liquid region from the normal melting point near 189 K to 553 K (ITS 90 temperature scale). These recommendations are based on the best available thermal conductivity data, obtained with transient hot-wire instruments incorporating bare and coated hot-wire cells with different geometries. The recommendations are considered consistent with radiation-free measurements of the fluid thermal diffusivity from light scattering, even at the highest temperatures. This correlation for liquid toluene extends the temperature range of the recommendations and is useful for evaluating the performance of high-temperature instruments.

The proposed value of the thermal conductivity of toluene at 298.15 K has an estimated uncertainty of 0.0008 W m⁻¹ K⁻¹, or 0.6%. The proposed tabulated data have estimated uncertainties of 1% for 189 K < T < 440 K, 1.5% for 440 K < T < 480 K, and 2% for 480 K < T < 553 K.

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